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METHODS OF SAMPLING AND TEST FOR ORGANIC PIGMENTS FOR PAINTS

(*First Revision*)

First Reprint MARCH 1996
(Incorporating Amendment No. 1)

UDC 667.622.3 : 620.113

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 6

September 1978

Indian Standard

METHODS OF SAMPLING AND TEST FOR ORGANIC PIGMENTS FOR PAINTS

(*First Revision*)

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Indian Standard

METHODS OF SAMPLING AND TEST FOR ORGANIC PIGMENTS FOR PAINTS

(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 12 February 1978, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

0.2 Organic pigments constitute the most important raw material in the formulation of paints, printing inks, rubber and plastics industry. The demand for various kinds of organic pigments is steadily increasing with development of these industries. At present a wide range of organic pigments is being manufactured in the country. The formulation of this standard was taken up with a view to introducing uniform practice in sampling and tests for organic pigments in the country.

0.3 This standard was first published in 1965. In this revision methods of test for sieve analysis, comparison of colour and staining power on reduction, fastness to light, bleeding and heat stability have been modified. A new method based on use of a miniature sandmill for comparison of colour and strength of coloured pigments has also been prescribed in addition to automatic muller method. The basic consideration in the preparation of this revision has been to see that every test method is based on actual experience under Indian conditions.

0.4 For residue on sieve, Indian Standard sieves (conforming to IS : 460-1962*) are prescribed. Where IS Sieves are not available, other equivalent standard sieves as judged by aperture size may be used.

0.5 Test methods not prescribed in this standard and not specified in the standard specification for an individual material, are subject to agreement between the purchaser and the supplier.

0.5.1 This standard is a necessary adjunct to material specification for organic pigments.

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*Specification for test sieves (*revised*).

0.6 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes methods of sampling and test for organic pigments for paints.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 1303-1963† and the following, shall apply.

2.1.1 *Approved Sample* — The sample accepted by the indentor or inspection authority as the basis for supply. When a sample is tested and approved by the purchaser or inspection authority, the result of such tests as permit the supplier to meet the limits imposed by the specification for deliveries shall be made available to the supplier.

2.1.2 *Tender Sample* — The sample submitted by the supplier with his tender.

2.1.3 *Standard Atmospheric Conditions for Testing* — An atmosphere with relative humidity of 63 ± 2 percent and temperature of 27 ± 2°C provided that in a given series of experiments, temperature does not vary by more than ± 1°C (*see also* IS : 196-1966‡).

2.1.4 *Room Temperature* — A temperature between 21°C and 38°C, unless otherwise specified.

2.1.5 *Freedom from Lead* (*Lead-Free)* — It means that the material does not contain lead or compounds of lead or a mixture of both calculated as metallic lead (Pb), exceeding 0 03 percent by mass.

3. SAMPLING

3.1 General Requirements of Sampling

3.1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

3.1.1 Samples shall not be taken in an exposed place.

3.1.2 The sampling implement and the containers for samples shall be made of metal or opaque glass on which the material has no action. They shall be clean and dry.

---

*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to paints (*revised*).

‡Atmospheric conditions for testing (*revised*).

3.1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling implement and the containers for samples from adventitious contamination.

3.1.4 The sample containers shall be of such size that they are almost but not completely, filled with the sample.

3.1.5 The sample containers shall be sealed airtight after filling and marked with full identification particulars, such as the date of sampling, the month and year of manufacture of the material, name of the sampler and any other relevant particulars of the consignment.

3.1.6 Samples shall be stored in such a manner as to avoid excessive variations of temperature.

3.2 Sampling Implement — Sampling shall be carried out by means of a suitable sampling scoop of the type shown in Fig. 1. The scoop shall be made of metal and shall have a semicircular (or C-shapcd, if found suitable) cross section.

3.3 Scale of Sampling

3.3.1 *Lot* — All the containers of the same size in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and of the same size shall be grouped together and each such group shall constitute a separate lot.

3.3.2 For ascertaining the conformity of the lot to the requirements of any specification, tests shall be carried out for each lot separately. The number of containers *n* to be selected from a lot of size *N* shall be in accordance with Table 1.

TABLE 1 SCALE OF SAMPLING FOR ORGANIC PIGMENTS

LOT SIZE N (1)	SAMPLE SIZE n (2)
Up to 60	2
61 „ 150	3
151 „ 300	4
301 „ 500	5
501 „ 1 000	6
1 001 „ 1 500	7
1 501 „ 2 000	8
2 001 „ 3 000	9
3 001 „ 4 000	10
4 001 „ 6 000	11
6 001 and above	12

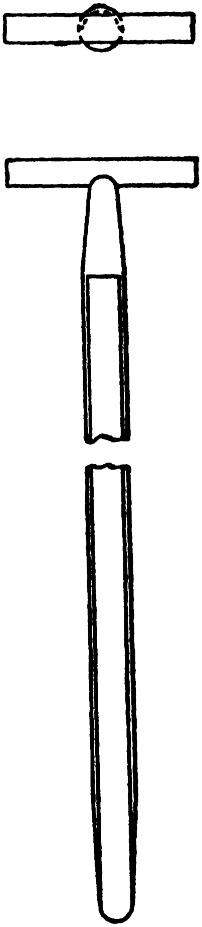


FIG. 1 SAMPLING SCOOP

3.3.3 The containers shall be selected at random and in order to ensure randomness of selection, a random number table* shall be used. In case such a table is not available, the following procedure is recommended for use:

> Starting from any container in the lot, count them in one order as 1, 2, 3,............., etc, up to *r* and so on where *r* is the integral part of *N/n* (*N* being the lot size and *n* the corresponding sample size given in Table 1). Every *r*th container thus counted shall be withdrawn to give sample for tests.

**See* IS : 4905-1968 Methods for random sampling.

3.4 Preparation of Test Samples

3.4.1 From each of the containers selected according to **3.3.3** a small representative portion of the material shall be drawn with the help of the sampling scoop by inserting it into the material at different portions.

3.4.2 Out of the material drawn from individual containers, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient for carrying out triplicate determinations for all the characteristics. If the quantities of material mixed from different portions are quite large, the process of coning and quartering may be adopted to obtain the requisite quantity (*see* Note). The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

NOTE — On a clean impervious surface, heap the samples into a cone by depositing separate small quantities one on top of the other, ensuring that the apex always remains in the same vertical line. Break down the cone, mix and reform the cone twice in the same manner. Flatten the third cone by pressing with a metal or glass sheet so that a mass of uniform thickness and diameter is obtained, and then quarter along two diameters. Discard the diagonally opposite quarters and heap remaining two quarters into a cone. Repeat this process until a sufficient quantity is left to provide a sample of not less than 150 g.

3.4.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers in the sample shall be for purchaser, another for the supplicr and the third shall be reserved as referee set.

3.4.4 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed airtight with stoppers and labelled with full identification particulars given in **3.1.5.**

3.4.5 The referee samples, consisting of a composite sample and a set of *n* individual samples, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two and shall be used in case of any dispute.

3.5 Number of Tests — Tests for determination of all characteristics shall be conducted on the composite sample. However, in case of doubt regarding uniformity of the material in a particular lot, all tests shall be conducted on all individual samples.

3.6 Criteria for Conformity — The lot shall be declared as conforming to the requirements of the specification if all the test results obtained after testing according to **3.5** satisfy the relevant requirements of the specification.

4. QUALITY OF REAGENTS

4.1 Unless otherwise specified, pure chemicals and distilled water (*see* 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

5. DETERMINATION OF SULPHATED ASH

5.1 Procedure — Cool a previously ignited and weighed crucible in a desiccator and weigh accurately into it about 2 g of the test sample. Heat gently in the flame of a Bunsen burner until burning and charring is complete. Add a few drops of concentrated nitric acid, heat gently again and cool. Moisten with a few drops of concentrated sulphuric acid and heat gently until fuming ceases. Ignite strongly and cool in a desiccator. Repeat ignition until constant mass is obtained.

3.2 Calculation

$$\text{Sulphated ash, percent by mass} = \frac{100\, m}{M}$$

where

m = mass in g of the ash, and

M = mass in g of the sample taken for the test.

6. DETERMINATION OF VOLATILE MATTER

6.1 Procedure — Weigh, to the nearest milligram, about 2 g of the test sample and spread it in a uniform layer on the bottom of a dry, tared wide-mouth cylindrical glass weighing bottle (flat bottomed, about 30 mm in height and about 70 mm in diameter) provided with a ground glass stopper. Heat the bottle and contents, with the stopper removed, in an oven at 100 ± 2°C for 2 hours. Insert the stopper, cool the bottle in a desiccator, momentarily release the stopper immediately before weighing and weigh to the nearest milligram. Repeat heating and cooling until constant mass is obtained.

6.2 Calculation

$$\text{Matter volatile, percent by mass} = \frac{100\,(M_1 - M_2)}{M_1}$$

where

M_1 = initial mass in g of the sample taken for the test, and

M_2 = final mass in g of the sample obtained after heating.

---

*Specification for water for general laboratory use (*second revision*).

7. DETERMINATION OF RESIDUE ON SIEVE

7.1 Apparatus

7.1.1 *Test Sieves* — 125-micron IS Sieve, 63-micorn IS Sieve and 45-micron IS Sieve (conforming to IS : 460-1962*), 50 to 100 mm diameter, with sides of 50 mm.

7.1.2 *Glass Rod* — tipped with rubber having hardness of 50 IRHD.

7.1.3 *Filter Crucible of Sintered Glass* — porosity grade P 40 (diameter of pores 16 to 40 µm).

7.1.4 *Water Supply* — at a pressure equivalent to a head of water 1 m.

7.1.5 *Rose* — as shown in Fig. 2.

7.2 Procedure

7.2.1 Weigh into a beaker, to the nearest 0.1 g, between 50 and 500 g (*see* Note 1) the test sample previously dried in accordance with **6**. Disperse the pigment (*see* Note 2) in a suitable quantity of water (usually of the order of 500 to 600 ml), using the rubber-tipped glass rod only to assist in the dispersion and pour the dispersion through the appropriate sieve (**7.1.1**). Return the residue retained on the sieve to the beaker and repeat the dispersion (without adding any further dispersing agent), using a similar quantity of water; pour onto the sieve as before. Repeat this operation once more and wash out the residue remaining in the beaker with water, using the glass rod if necessary.

7.2.2 Wash the residue on the sieve, using the rose fitted to the constant-head water supply. Wash the residue into the previously tared sintered glass crucible and dry at 105 ± 2°C. Cool in a desiccator and weigh to the nearest 0.1 mg. Repeat heating, cooling and weighing operations until constant mass is obtained.

NOTE 1 — The mass of pigment chosen shall be such that, wherever possible, the expected sieve residue is about 0.2 g. With the finest pigments, samples up to 500 g shall be used.

NOTE 2 — If necessary, a suitable wetting or dispersing agent may be used, such as ethanol or a condensed naphthalene sulphonate.

7.3 Calculation

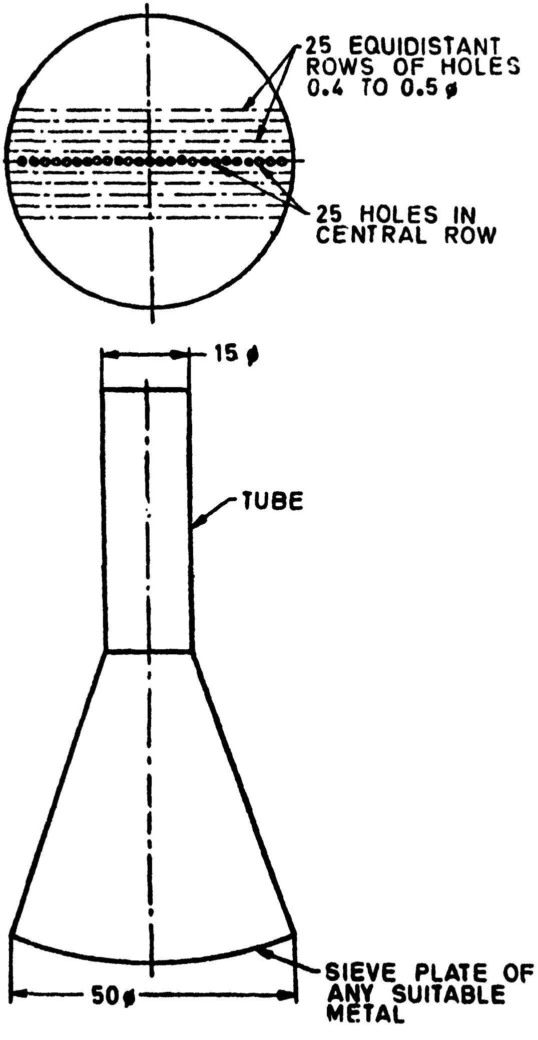
$$\text{Residue on sieve, percentage by mass} = \frac{100\, m_2}{m_1}$$

where

m_1 = mass in g of the test portion, and
m_2 = mass in g of the residue.

---

*Specification for test sieves (*revised*).


25 EQUIDISTANT ROWS OF HOLES 0.4 TO 0.5 ϕ
25 HOLES IN CENTRAL ROW
15 ϕ
TUBE
SIEVE PLATE OF ANY SUITABLE METAL
50 ϕ

All dimensions in millimetres.

FIG. 2 Rose USED FOR WASHING THE RESIDUE OF SIEVE

8. DETERMINATION OF MATTER SOLUBLE IN WATER AND *p*H VALUE OF AQUEOUS EXTRACT

8.1 Materials

8.1.1 *Buchner Funnel* — 10 cm diameter.

8.1.2 *Filter Papers* — Whatman No. 5 and 42 or equivalents.

8.1.3 *Barytes* — good quality, thoroughly washed with boiling distilled water and dried.

8.2 Procedure — Weigh accurately about 10 g of the test sample and wet thoroughly with a mixture of 5 ml of rectified spirit (*see* IS : 323-1959*) and 5 ml of water. Add 150 ml of water and boil for 5 minutes. Cool the mixture to room temperature. Filter the suspension through a double, hard, close texture filter paper (Whatman No. 5 or equivalent) using a small Buchner funnel. Collect the filtrate in a clean flask and when it is running clear, replace the collecting fiask by a second clean flask, returning the first portion of the filtrate collected in the first flask to the main suspension for refiltering. Wash out the first flask and the vessel in which the suspension was prepared with approximately 30 ml of distilled water using the washings also to wash the residue of pigment on the filter. Press the residue of pigment on the filter well to avoid channelling. Dilute the collected filtrate and washings to 250 ml with water and shake well. Evaporate 100 ml of the filtrate to dryness in a previously dried and weighed silica dish. Dry the residue so obtained to constant mass in an oven at 100 ± 2°C.

8.2.1 In case of pigments which pass through the filter paper, filtration shall be carried out through barytes bed and then proceed as prescribed in **8.2**. The barytes bed shall be prepared as given in **8.2.1.1.**

8.2.1.1 Weigh about 30 g of the washed barytes in a beaker and make a slurry with water. Transfer the slurry to the Buchner funnel on which a Whatman No. 42 filter paper (or equivalent) is already placed. Filter the slurry under vacuum till all the water is drained off and a uniform bed is obtained.

8.3 Calculation

$$\text{Matter soluble in water, percent by mass} = 250\,\frac{m}{M}$$

where

m = mass in g of the residue obtained after evaporation, and
M = mass in g of the sample taken for the test,

---

*Specification for rectified spirit (*revised*).

8.4 *p*H Value of Aqueous Extract — Determine the *p*H value of the filtrate obtained under **8.2** by a *p*H meter.

9. DETERMINATION OF OIL ABSORPTION VALUE

9.0 Outline of the Method — Linseed oil is added to the material in small portions, thoroughly grinding the material after each addition, until the mass is coherent under pressure of the palette knife but does not smear the plate.

9.1 Apparatus

9.1.1 *Ground-Glass or Marble Plate* — At least 30 × 40 cm.

9.1.2 *Steel Palette Knife* — A suitable knife is one having a tapered blade 140 to 150 mm long, 20 to 25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point.

9.1.3 *Burette* — 25-ml, graduated in 0.1 ml divisions (conforming to IS : 1997-1967*).

9.2 Material Required

9.2.1 *Acid Refined Linseed Oil* — conforming to IS : 75-1973† and having an acid value of 7.5 to 8.5.

9.3 Procedure — Weigh accurately about 5 g of the test sample and place it on the ground-glass or marble plate. Add linseed oil slowly, 5 drops at a time, from the burette. After each addition of 5 drops, thoroughly grind the oil with the pigment using the palette knife and continue addition of the oil at this rate until conglomerates of pigment and oil are formed. From this point, add the oil one drop at a time and follow each addition of oil by thoroughly running with the palette knife. Cease the addition of oil when the last drop of oil causes conglomerates to collect together into a single mass which is coherent under the pressure of the palette knife and which shall just not smear the plate. The time taken for the whole operation of the test shall be 20 to 25 minutes, and during this time, the whole of the pigment mass shall be manipulated with the application of maximum effort by the operator.

9.4 Calculation

9.4.1 Oil absorption value on a mass/mass basis $= \frac{93\,V}{M}$

9.4.2 Oil absorption value on a volume/mass basis $= \frac{100\,V}{M}$

where

V = volume in ml of the acid-refined linseed oil required, and
M = mass in g of the sample taken for the test.

---

*Specification for burettes *(first revision)*.

†Specification for linseed oil, raw and refined (*second revision*).

10. COMPARISON OF COLOUR

10.0 Outline of the Method — The material under test is rubbed out by automatic muller and applied on a glass plate to compare the colour with that of an approved sample.

10.1 Apparatus

10.1.1 *Automatic Muller* — an apparatus consisting of two circular horizontal ground-glass plates, the lower one of which can be revolved in contact with the top plate. The pigment and the medium mixture is placed between the plates, force applied by means of weights and the lower plate revolved for a desired number of revolutions (*see* Notes 1 and 2).

NOTE 1 — As one plate revolves over the other plate, the paste which is being ground will receive much less grinding at the centre and more of it away from the centre. In order to minimize this variable in testing, draw two concentric circles under the base plate of the muller in such a way that they are visible through the plate. These may be drawn either on a paper which is then inserted under the base plate, or directly on the bottom of the plate. The diameter of the two circles is 10 cm and 15 cm respectively.

NOTE 2 — In the case of Rathi s muller, the grinding surface of the plates is 25 cm in diameter. The weights which can be applied for grinding are between 25 kg and 100 kg. The pressure per unit area applied under different weights is given below. In case of other mullen where the grinding surfaces are of different dimensions and the weight which can be added to the muller are also different, grinding should be done under such conditions that the pressure per unit area is near to that obtained in the case of Rathi's muller. Figures for pressures obtained in the case of a Hoover or Ault and Wiborg muller, which have a grinding surface of 18.5 cm in diameter and to which weights of 25 kg, 50 kg and 75 kg can be applied are also given below:

Weight Applied kg	*Pressure Developed,* kg/cm^2	
	Rathi's Muller (Plate Dia 25 cm)	Hoover/Ault & Wiborg (Plate Dia 18.5 cm)
25	0.05	0.08
50	0.10	0.17
75	0.15	0.25
100	0.20	—

10.1.2 *Stainless Steel Palette Knife* — A suitable knife is one having a tapered blade 140 to 150 mm long, 20 to 25 mm wide at its widest point and not less than 12.5 mm at its narrowest point.

10.1.3 *Glass Plate* — 150 × 50 mm, clear and colourless.

10.1.4 *Scraper* — About 7 × 3 cm and having one sharp edge.

10.1.5 *Burette* — 10 ml, conforming to IS : 1997-1967*, with a delivery so that 35 drops or equal to 1 ml of linseed stand oil.

10.1.6 A film applicator for applying a wet film of 50 to 100 microns.

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*Specification for burettes (*first revision*).

10.2 Materials Required

10.2.1 *Linseed Stand Oil* — conforming to type 3 of IS : 79-1975*.

10.2.2 *Standard White Pigment Paste* — having following composition:

a) Titanium dioxide (anatase)	90 parts
b) Heat-treated linseed stand oil	59.5 parts
c) Cobalt naphthenate (cobalt content about 9 percent by mass)	0.2 part
d) Lead naphthenate (lead content about 25 percent by mass)	0.3 part

NOTE — The paste is ground on a triple roll mill to minimum 7 on Hegmann gauge 0.005.

10.3 Procedure

10.3.1 Pigments are divided in following groups for taking the quantity of pigment and medium for the test. Organic pigments are covered in Group 1 and carbon black in Group 2.

Group 1 — 2 ± 0.001 g pigment and 3 ± 0.005 g linseed stand oil.

Group 2 - 1 ± 0.001 g pigment and 5 ± 0.005 g of linseed stand oil.

10.3.2 Weigh the requisite quantity of the pigment as given in **10.3.1** and counterbalance it with the approved sample of the pigment. Transfer the weighed sample to the clean lower glass plate of the muller. Add the requisite quantity of the stand oil from the burette, mixing it with the knife. Allow the burette to drain to its true level (about 2 minutes). Mix it to a smooth paste and spread it evenly in the 2.5 cm band between the two circles on the bottom plate. Transfer any paste still sticking to the palette by drawing it across the middle portion of the upper plate. Close the muller. Add additional weights to exert a pressure of 0.2 kg/cm^2 on the bottom plate. Run the muller for five cycles of 50 revolutions each. After each rubbing cycle, scrape the paste from each mulling surface and respread over the same 2.5 cm band area and continue mulling. When grinding is completed, mix the paste thoroughly with the palette knife and store it on the palette.

10.3.3 Immediately prepare a paste from the approved sample in an exactly similar manner.

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*Specification for linseed stand oil for paints *(first revision)*,

10.3.4 Apply one daub each from these two pastes, about 2.5 cm wide and 4.0 cm long, on the glass panel. The edges of the daubs from the two samples should be touching each other. Draw the scraper lightly over the pastes to even off the ridges, so that these can be observed from an even plane. Compare the colours immediately in diffused north daylight observing from the top.

NOTE — The consistency of the pastes is such that these can be just thrown from the palette knife by a gentle whipping motion of the hand or by gentle tapping of the palette knife with a finger.

11. COMPARISON OF STAINING POWER AND COLOUR ON REDUCTION

11.0 General — Two methods have been prescribed. The first one makes use of automatic muller whereas the second one uses a miniature sandmill.

11.1 Automatic Muller Method

11.1.0 *Outline of the Method* — Pastes are prepared from test sample and an approved sample by automatic muller method and compared.

11.1.1 *Procedure* — Weigh 5 g of the standard white pigment paste as specified in **10.2.2** and as much of the coloured paste (*see* **10.3.1**) as required for reduction in various ratios, namely, 1:1, 1:5, etc. Table 2 gives the quantity of coloured paste required to get a particular ratio.

TABLE 2 QUANTITY OF COLOURED PASTE REQUIRED FOR COLOUR REDUCTION

GROUP	PIGMENT BINDER RATIO	PERCENTAGE OF PIGMENT	QUANTITY OF COLOURED PASTE IN g TO BE WEIGHED IN ORDER TO ACHIEVE THE RATIO OF COLOURED PIGMENT/WHITE PIGMENT		
			1:1	1:5	1:10
(1)	(2)	(3)	(4)	(5)	(6)
1	2 : 3	40	—	1.5	0.75
2	1 : 5	16.67	—	—	1.8

11.1.2 Mix the two pastes and rub on the muller for 4 × 25 revolutions, under a pressure of 0.05 kg/cm^2, as described in **10.3.2**. Apply the reduction pastes on a glass plate in 50 to 100 microns wet film thickness with an applicator and compare the colour and strength by examining the test panel with that from the approved sample similarly prepared, in diffused north daylight from the top immediately after application.

11.2 Miniature Sandmill Method

11.2.0 *General* — This method prescribes determination, through the use of a miniature sandmill, of colour and strength of dry colour pigments.

11.2.1 *Apparatus*

11.2.1.1 *Balance* — Sensitive to 10 mg with a capacity in excess of 300 g.

11.2.1.2 *Miniature sandmill* — A laboratory disperser equipped with around 40 mm fibre rotary disc impeller rotating at a constant 8 000 rev/min under varying load conditions. The shaft upon which the impeller is mounted shall be sufficiently balanced so that no whip of the shaft is observed between 0 and 10 000 rev/min.

11.2.1.3 *Cylinder* — 100 ml, graduated.

11.2.1.4 *Beakers* — 200 ml, tall-form, glass, stainless steel, or polyethylene (approximately 60 mm in inside diameter).

11.2.1.5 *Strainers* — paper cone, disposable, about 425 micron.

11.2.1.6 *Fibre discs* — phenolic laminated, 40 mm diameter and 6 mm thick.

11.2.1.7 *Paper charts* — smooth, surface-coated, the surface of which should be impervious to paint liquids.

11.2.1.8 *Film applicator* — with an 0.2 mm clearance and at least 76 mm wide.

11.2.2 *Material*

11.2.2.1 *Approved sample of pigment*

11.2.2.2 *Sand* — regular 850 to 600 μm cement testing sand can be used but it is recommended that the sand be screened to remove all but 600 µm particles. Glass beads of 850 to 600 μm may also be used instead of sand.

11.2.2.3 *Grind vehicle* — a long oil alkyd resin reduced to 47 percent solids with petroleum hydrocarbon solvent.

11.2.2.4 *White tinting paint* — a flat white tinting paint compatible with the dispersion vehicle meeting the following requirements:

a) *Gloss (60 degrees)* — less than 4; and
b) *Contrast ratio* — minimum 99.2 (51 μm dry film).

11.2.2.5 *Drier blend* — 1 part of 6 percent manganese naphthenate, 2 parts of 6 percent cobalt naphthenate, and 4 parts of 24 percent lead naphthenate by mass.

11.2.3 *Procedure for Mass Colour*

11.2.3.1 Weigh to the nearest 10 mg an appropriate amount of pigment to be tested into a 200-ml tall-form beaker. To the pigment in the beaker carefully weigh in 55.00 ± 0.01 g of the long oil alkyd grinding vehicle. Then add 60 ml of sand from a graduated cylinder and 1.00 ± 0.01 g of the drier blend. Stir the sand, vehicle and pigment with a spatula until the pigment is completely wetted.

11.2.3.2 Carefully clamp the beaker on the miniature sandmill and adjust to the height of the disc so that it is between 19 to 25 mm above the bottom of the beaker. Turn on the mill and grind for exactly 10 minutes unless otherwise agreed upon, at a shaft speed of 8 000 rev/min, accurately timing the length of the grind with a stop-watch or other suitable timing device.

11.2.3.3 Shut off the mill and raise the impeller until it is just clear of the liquid in the beaker and turn the mill on again for a few seconds to remove excess mill base from the impeller.

11.2.3.4 Remove the sample from the mill and filter into a suitable container through a cone strainer or equivalent to 420 µm disposable screen.

11.2.3.5 Repeat the procedure with approved pigment.

11.2.3.6 Draw the sample and approved sample down simultaneously on a paper chart over a vacuum drawdown plate or other suitable plane surface using the application bar specified in **11.2.1.8.** Compare the colour visually while still wet and then set aside in a dust-free area to dry.

11.2.4 *Procedure for Tint Colour*

11.2.4.1 From the receiving container used in **11.2.3** weigh 5.000 ± 0.010 g of the strained mill base into a 200-ml beaker and add 100.00 ± 0.05 g of white tinting paint.

11.2.4.2 Place the beaker on the mill (without sand) and mix at 8 000 rev/min for 5 minutes.

11.2.4.3 Repeat the above procedure with the approved sample and then proceed as prescribed in **11.2.3.6.**

12. COMPARISON OF FASTNESS TO LIGHT

12.0 Outline of the Method — Pastes of the material under test and the approved sample are prepared using linseed stand oil or medium as agreed between the purchaser and the supplier. They are applied on cardboard pieces which are subjected to the action of ultraviolet light in a fadeometer for the same period and then compared with each other for change in colour.

12.1 Apparatus

12.1.1 *Cardboard Pieces*

12.1.2 *Twin Applicator* — block type.

12.1.3 *Fadeometer* — The essential features of which are given below:

a) *Source of light* — A carbon arc composed of carbon electrodes and enclosed in a heat-resisting glass globe to produce a complete spectrum from 279 nm to 1 200 nm.

The arc should operate on a direct current of approximately 11 to 13 A or on alternating current of 50 to 60 Hz and approximately 13 to 17 A, with 110 to 140 V across the arc.

b) The test specimens are vertically hung in test racks on the inner wall of a cylinder (rotating or fixed type), concentric with the arc. The distance of the test specimens shall be approximately 250 mm from the arc. No part of the specimens shall be more than 100 mm above or below the horizontal plane passing through the centre of the arc when it is fitted with new electrodes.

c) The air inside the chamber shall be humidified by placing water in a trough or water soaked cotton wicks in the chamber. A fan or blower unit keeps a flow of air over the specimens and through the chamber.

d) A black panel thermometer placed in a rack in the chamber shall record a temperature not more than 74°C.

12.1.3.1 In this test, xenon arc fadeometer may also be used.

12.1.4 *Ground-Glass or Marble Plate* — at least 30 × 40 cm.

12.1.5 *Steel Palette Knife* — A suitable knife is one having a tapered blade 140 to 150 mm long, 20 to 25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point.

12.1.6 *Burette* — 10 ml, graduated in 0.1 ml divisions (conforming to IS : 1997-1967*).

12.1.7 *Geometric Grey Scale* — conforming to IS : 768-1956†.

12.1.8 *Cover Sheet* — a sheet of aluminium foil or other suitable opaque material.

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*Specification for burettes (*first revision*).

†Method for evaluating change in colour.

12.2 Materials Required

12.2.1 *Linseed Stand Oil* — conforming to Type 3 of IS : 79-1975*.

12.2.2 *Naphthenate Driers* — lead naphthenate, having 19 ± 1 percent lead and cobalt naphthenate, having 6.0 ± 0.5 percent cobalt, in solution in petroleum hydrocarbon solvent, 145/205 (low aromatic), conforming to IS : 1745-1966†.

12.2.3 *Petroleum Hydrocarbon Solvent* — 145/205 (low aromatic), conforming to IS : 1745-1966†.

12.2.4 *Zinc Oxide* — conforming to IS : 35-1975‡.

12.3 Procedure

12.3.1 *Full Strength* — Weigh, to the nearest milligram, 0.2 g of the test sample and place on the ground-glass or marble plate. Add linseed stand oil, 4 to 5 drops at a time, rubbing the oil into the pigment with the palette knife after each addition until a smooth paste is obtained as prescribed in **10.3.2,** the volume of oil to be added is such that the pigment volume is 20 percent. If necessary add a small volume of petroleum hydrocarbon solvent to case mixing.

12.3.2 *Reduced Strength* — Weigh, to the nearest milligram, 2.0 g of zinc oxide and 0.02 g of the test sample and place on the ground-glass or marble plate. Add linseed stand oil, 4 to 5 drops at a time, rubbing the oil into the pigment with the palette knife after each addition until the maximum strength of the pigment has been developed, and the volume of oil added is four times the volume of the zinc oxide.

12.3.3 At the final stage of both **12.3.1** and **12.3.2,** add the naphthenate drier, so that the amount in the final paint is 1.0 percent of lead and 0.1 percent of cobalt, calculated as metals on the oil. Alternatively, the linseed stand oil may be treated before use with the appropriate naphthenates to give 1.0 percent of lead and 0.1 percent of cobalt on the oil, and this blended medium used throughout the test. Treat the approved sample in the same way.

12.3.4 By means of the twin applicator, with shims set to give a wet thickness of 0.08 mm, apply to the cardboard pieces, films of the dispersion of the test sample and approved sample. Allow the films to dry in a horizontal position for 24 hours in diffused daylight at normal room temperature. Mount the strips of pigment-coated cardboard on a suitable support (for example, a piece of wood) and cover the middle one third of both films with the cover sheet, clipping down the edges to prevent

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*Specification for linseed stand oil *(first revision*).

†Specification for petroleum hydrocarbon solvents *(first revision*).

‡Specification for zinc oxide for paints *(first revision*).

buckling. Expose the assembly to the source of light in a fadeometer and at suitable intervals, examine them by raising the cover sheet. Replace the cover exactly in its original position and continue the exposure until the contrast produced on the exposed and unexposed positions of the approved sample is equal to Grade 4 on the geometric Grey Scale. At this point compare the test sample and approved sample and record the result.

12.3.5 Then fix a further cover sheet so that two-thirds of the films (including the original one-third) are covered and continue the exposure until the fully exposed and completely unexposed portions of the approved sample show a contrast equal to Grade 3 of the Geometric Grey Scale. Then assess the comparative behaviour of the test sample with the approved sample.

13. TEST FOR BLEEDING

13.0 General — Bleeding of pigments in solvents or media at ambient temperature, at higher temperature (at 60°C) and in over-coating composition has been prescribed.

13.1 Apparatus

13.1.1 *Cardboard Pieces*

13.1.2 *Twin Applicator* — block type.

13.1.3 *Ground-Glass or Marble Plate* — at least 30 × 40 cm in size.

13.1.4 *Steel Palette Knife* — A suitable knife is one having a tapered blade 140 to 150 mm long, 20 to 25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point.

13.2 Materials Required

13.2.1 *Dispersing Medium* — Which may contain drying oils, resins, plasticizers, solvents, etc, of such a nature and in such amounts as may be appropriate and agreed between the purchaser and the supplier.

13.2.2 *Over-Coating Composition* — as agreed between the purchaser and the supplier.

13.3 Procedure

13.3.1 *Bleeding Test at Room Temperature* — Take approximately 0.5 g of the pigment under test. Prepare a cone out of an ordinary round filter paper of about 10 to 11 cm in diameter. Open out the cone by having on one side one layer and on the other side three layers. Put the pigment under test into the cone. Fold the cone with the pigment three times more so that it can go into a glass test tube. Tie the filter paper cone, containing the pigment, at the top with a string and suspend it at ambient temperature in a test tube containing about 10 to 15 ml of solvent/medium as agreed between the supplier and the purchaser. After 24 hours remove the filter paper cone and examine the solvent/medium for any discolouration. Carry out the test with the approved sample side by side in a similar way and compare the results. The colour of the solvent/medium used for sample under test should not be deeper than that with the approved sample.

13.3.2 *Bleeding Test at Higher Temperature (60°C)* — Take approximately 0.5 g of the pigment under test. Prepare a cone out of an ordinary round filter paper of about 10 to 11 cm in diameter. Open out the cone by having on one side one layer and on the other side three layers. Put the pigment under test, into the cone. Fold the cone with the pigment three times more so that it can go into a glass test tube. Tie the filter paper cone, containing the pigment, at the top with a string and suspend it in a test tube containing about 10 to 15 ml of solvent/medium as agreed to between the purchaser and the supplier, kept on a water-bath at 60°C. After 1 hour, remove the filter paper cone and examine the solvent/medium for any discolouration. Carry out the test for the approved sample side by side in a similar way and compare the results. The colour of the solvent/medium used for sample under test should not be deeper than that with the approved sample.

13.3.3 *Bleeding Test in Over-Coating Composition* — Prepare a pigment dispersion of the test sample as prescribed in **10.3.2** in the agreed dispersing medium. Dilute this to an appropriate consistency by further addition of a suitable medium or thinner. Treat the approved sample in the same way. Apply the mixture of test sample and approved sample to a piece of cardboard in touching parallel strips, not less than 25 mm wide and 100 mm long. Allow them to dry. Apply a coat of the over-coating medium over the test strips by the method agreed between the purchaser and the supplier in such a manner that complete obscuration is obtained, and allow to dry. The drying period of the coloured coats and the over-coating shall be as agreed between the purchaser and the supplier and may include a period of stoving. Examine the test strips after 30 minutes and after 48 hours for evidence of bleeding as revealed by differences in colour of the top coat (overcoat) over both coloured strips.

14. TEST FOR HEAT STABILITY

14.0 Outline of the Method — The test sample and the approved sample are dispersed separately in a dispersing medium as agreed between the purchaser and the supplier and after adjusting the consistency, applied on a suitable substrate side by side by the film applicator at the same wet film thickness, baked at a temperature and time as agreed between the purchaser and the supplier, and then compared.

14.1 Apparatus

14.1.1 *Test Panels* — bright tin plate or light gauge mild steel panels 5 × 10 cm in size, the surface of which have been cleaned and lightly abraded.

14.1.2 *Twin Applicator* — block type.

14.2 Material Required — Dispersing medium which may contain drying oils, resins, plasticizers, solvent, etc, of such a nature and in such amounts as may be appropriate and agreed between the purchaser and the supplier.

14.3 Procedure — Weigh about 5 to 10 g of the test sample and disperse in an agreed dispersing medium (using thinner, if necessary). Prepare identically the dispersion of the approved sample in a similar manner. Apply on a panel touching parallel strips of the two dispersions side by side with a twin applicator of the same wet film thickness. Bake the panel for the temperature and time as agreed between the purchaser and the supplier and after baking, allow to cool and make comparison in normal daylight. The test sample should not show any greater change of colour as compared with the approved sample.

15. DETERMINATION OF RESISTANCE TO ACID, ALKALI AND LIME

15.0 Outline of the Method — Resistance to acid, alkali and lime is determined by finding out the degree of bleeding of the solutions prepared from material under test and the approved sample in hydrochloric acid, sodium hydroxide and calcium hydroxide solution respectively. Additionally the treated pigment is washed, dried and tested against pigment for full colour and reduction as prescribed in **10** and **11.**

15.1 Reagents

15.1.1 *Standard Hydrochloric Acid* — 0.5 N (*see* IS : 265-1962*).

15.1.2 *Standard Sodium Hydroxide Solution* — 0.25 N.

15.1.3 *Calcium Hydroxide Solution* — 10 percent (*m/v*).

15.2 Procedure

15.2.1 *Resistance to Acid* — Take about 1 g of the pigment in a 100-ml flask and shake with about 50 ml of standard hydrochloric acid. Allow the contents to stand for one hour. In another flask treat an equal quantity of the approved sample in a similar manner. Note the colour change in the supernatant liquid of the material under test as compared

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*Specification for hydrochloric acid (*revised*).

with that of the approved sample. Wash the pigment free from acid, filter and dry in an oven at 100 ± 2°C. Determine the change in colour and staining power of the pigment as prescribed in **10** and **11**. Compare the change in the test sample with that obtained in the case of the approved sample, when similarly treated and tested.

15.2.2 *Resistance to Lime* — Repeat the above test (*see* **15.2.1**) but using calcium hydroxide solution in place of standard hydrochloric acid.

15.2.3 *Resistance to Alkali* — Repeat the above test (*see* **15.2.1**) but using standard sodium hydroxide solution in place of hydrochloric acid.

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